Agriculture and Food

AIMS Agriculture and Food, 10(1): 40–57.
DOI: 10.3934/agrfood.2025003
Received: 26 November 2024
Revised: 06 January 2025
Accepted: 16 January 2025
Published:03 March 2025

https://www.aimspress.com/journal/agriculture

Research article

The physico-chemical characteristics of peel essential oils of sweet orange with respect to cultivars, harvesting times and isolation methods

Burcu Bozova[1], Muharrem Gölükcü[1], Haluk Tokgöz[1], Demet Yıldız Turgut[1], Orçun Çınar[1], Ertuğrul Turgutoglu[1] and Angelo Maria Giuffrè[2,*]

[1] Republic of Türkiye Ministry of Agriculture and Forestry, Batı Akdeniz Agricultural Research Institute [BATEM], Food Technology and Medicinal Aromatic Plants Department, 07112 Antalya, Turkey
[2] Department AGRARIA, University of Studies "*Mediterranea*" of Reggio Calabria, 89124 Reggio Calabria, Italy

* **Correspondence:** Email: amgiuffre@unirc.it; Tel.: +39-327-7022840.

Abstract: Within the scope of this study, essential oil content and its quality parameters of Batem Fatihi, Navelina, Washington Navel, Valencia Late, and Moro cultivars were evaluated with respect to harvest time and isolation methods. Essential oils were obtained using two methods (hydrodistillation and cold pressing) during four harvest periods of each cultivar. Physico-chemical characteristics of essential oils were evaluated, and the chemical components of the oil were identified via GC-MS/FID. Based on the hydrodistillation data, the essential oil ratios ranged between 1.69–2.85 % in samples. While the effects of cultivar and harvesting time on relative density, refractive index, and optical activity values were not statistically important, the effect of isolation method was important ($p < 0.05$). Relative densities of essential oils obtained by cold pressing were higher (0.8440) than those obtained by hydrodistillation (0.8402). Additionally, refractive index values of the samples obtained by cold pressing were higher (1.4739) than those obtained by the hydrodistillation method (1.4723). On the other hand, it was observed that the optical activity values of the samples obtained by hydrodistillation were higher (98.20°) than those obtained by cold pressing (95.32°). Chromatographic analysis indicated the presence of 10 compounds in the essential oil. Essential oil compositions also showed some differences on the basis of cultivars, harvesting time, and isolation methods, but these differences generally were not statistically significant. Additionally, there were some differences in determined components with respect to cultivar, harvesting time, and isolation method. The most important component in all analyzed samples was determined to be limonene and it was distributed between 94.9–96.4%. The second highest component was determined as β-myrcene

and ranged between 1.9–2.0%. While the effect of isolation method and harvesting time on limonene was not important ($p > 0.05$), the effect of cultivar was significantly important ($p < 0.05$). On the other hand, both cultivar and harvesting time effects on β-myrcene content were significantly important. Our results showed that there is a significant variation in some quality parameters of the orange peel essential oils according to cultivar and isolation methods.

Keywords: sweet orange; cultivar; peel essential oil; harvesting time; isolation technique

1. Introduction

Essential oils are generally obtained from the leaves, fruits, barks, or roots of plants, and are a natural product that is liquid at room temperature, can easily crystallize, is usually colorless or pale yellow, and has a strong and aromatic odor [1]. Essential oils consist of a complex mixture of pleasant-smelling and volatile components that are secondary plant metabolism products. Most of the components found in their structures are terpenoids, monoterpenes, and sesquiterpenes [2].

The production and consumption of essential oils have been increasing in recent years [3]. It is reported that the trade value of essential oils in the world is approximately 6 billion US dollars. It is stated that citrus peel oils are the most traded essential oils in the world [4]. In addition to its economic value, citrus peel oil production is very important in terms of waste evaluation. In fact, citrus peels generally appear as waste in fruit juice production. However, citrus peels are a rich source of functional components such as essential oil, pectin, and phenolic compounds [5–7]. A significant portion of essential oil production is carried out by the USA, China, and Brazil. Countries with high consumption are the USA, England, France, Japan, and Germany [8]. Türkiye, on the other hand, exports products such as rose essential oil, thyme essential oil, and bay laurel essential oil while importing some essential oils, especially citrus peel oils [9]. It is stated that there are more than 10,000 cultivars of citrus fruits in the world [10]. One of the most produced species among citrus fruits is orange [11]. Citrus peel oils can be obtained by cold pressing, hydrodistillation, and solvent extraction methods [12–16]. Citrus peel oils contain many bioactive components with different functional properties such as limonene, myrcene, α-pinene, β-pinene, sabinene, and linalool [17–19]. Citrus peel oils, which can be obtained by different methods, are used in many areas such as the food industry, cosmetics, perfumery, pharmaceutical industry, and the production of cleaning products [19–22]. In this sense, although synthetic products can also be used, the demand for products produced from natural sources is constantly increasing [23].

Türkiye realized 2.8% of the world's citrus production and 1.73% of the orange production in 2022 [24]. Considering the citrus production data, Turkey has great potential in the production of citrus peel oils. However, a significant portion of citrus oils is met through imports. According to TÜİK data, the total import value of citrus essential oils, one of the most imported essential oils, was 6,738,217 USD in 2020, while this value reached 15,641,898 USD in 2023. Considering the 2023 data, 53% of this is orange peel essential oil [25]. These data show that the production of these products is very important for the country's economy. Citrus essential oils are on the GRAS (Generally Recognized as Safe) list with their broad-spectrum biological activities being antimicrobial, antifungal, antioxidant, anti-inflammatory, and anxiolytic [20,26,27]. The most important feature of citrus peel essential oils is their high limonene content. It has been reported that the limonene content of citrus peel essential

oils (orange, mandarin, bergamot, bitter orange) is distributed in a very wide range, such as 36.54% to 96.10% [28]. It is stated that orange peel oils stand out with their high limonene content among citrus fruits [17]. Differences in the amount of limonene and other components of orange essential oil can be observed depending on the cultivars. Limonene (p-mentha-1,8-diene) is used in many areas such as food, medicine, and cosmetics on an industrial scale. Its use in the cosmetics and pharmaceutical industry is due to its aromatic properties as well as its high absorption. Limonene, which is widely used in the food industry due to its aromatic properties, is considered "generally recognized as safe" (GRAS) on the FDA list [29,30]. It has been reported that the composition of citrus peel oils may differ according to the type and cultivar as well as the isolation method (cold press, hydrodistillation) [31,32].

In Türkiye, which has significant potential in terms of raw materials, the production of such products is important for the world and national economy. Cultivars of Washington Navel, Navelina, and Valencia Late are the most common in Türkiye. In addition, the Moro cultivar is widely grown among blood oranges. The number of orange cultivars is constantly increasing though breeding studies. Batem Fatihi, which was used in the study, is a new cultivar developed as a result of breeding studies. There is a great interest in the physico-chemical properties of different parts of the citrus fruits. In addition, the evaluation of citrus peels, which are seen as waste, will also contribute to the development of the producer and processing industry. The citrus cultivars to be used in production, the harvest time of the material used, and the isolation method are among the determining factors on the quality of essential oil. Within the scope of the study, it was aimed to reveal some quality characteristics and essential oil compositions of peel essential oils obtained from 5 orange cultivars with two methods in four different harvest periods.

2. Materials and methods

2.1. Plant material

This research was carried out between 2021-2023 in the Aksu-central unit of the Batı Akdeniz Agricultural Research Institute (Antalya, Türkiye). Five orange (*Citrus sinensis* (L.) Osbeck) cultivars were used in the research. Each commercial cultivar was harvested in two production seasons (2021–2022, 2022–2023) covering four different harvest periods (Table 1). The materials used within the scope of the research were obtained from the citrus parcels of the Kayaburnu unit of the Batı Akdeniz Agricultural Research Institute. During the harvesting process, fruit samples were taken from four sides of each tree. The harvested fruit samples were brought to the Food Technology and Medicinal Plants Laboratory on the same day, and the analysis was started.

Table 1. Orange cultivars and harvest times.

Harvest	Batem Fatihi	Navelina	Washington Navel	Valencia Late	Moro
1	10 November	10 November	10 November	20 February	02 January
2	30 November	30 November	30 November	10 March	20 January
3	20 December	20 December	20 December	30 March	10 February
4	10 January	10 January	10 January	20 April	02 March

First, fruit weight and peel ratio were analyzed for the samples brought to the laboratory. For this purpose, 10 fruits were used for each repetition. Each fruit and its peels were weighed to an accuracy

of 0.01 g. Fruit weight and peel ratio were given by taking the average of the measurement values from ten fruits in each repetition.

2.2. Hydrodistillation (HD) process

Essential oil in the samples was extracted by the hydro-distillation method with a Clevenger-type apparatus according to ISO 6571 [33]. For this purpose, 200 mL of distilled water was added to 50 g of fresh fruit peel, homogenized with a blender (Waring 8011ES, USA), and then subjected to distillation using a Clevenger device (Isotex, Türkiye). The amount of essential oil is given by volume based on the weight of fresh fruit peel (mL/100 g, %).

2.3. Cold Press (CP) process

For cold pressing, essential oil was collected according to the Kırbaslar et al. [34]. For this purpose, the flavedo part of the fruit peels was grated, and then this part, which is rich in essential oil, was subjected to manual pressing with a 10 cm diameter seven-hole kitchen type apparatus. The water-essential oil emulsion was collected and then separated by centrifugation. This process was applied at 15294 × g at 20 °C for 20 minutes. The amount of essential oil is given by volume based on the weight of fresh fruit peel (mL/100 g, %).

2.4. Relative density, refractive index, and optical rotation

Physico-chemical characteristics are an important criterion of the quality and purity of essential oils. The obtained essential oils were analyzed for relative density, refractive index, optical rotation, and essential oil composition, which are among the basic quality analyzes specified in the European Pharmacopoeia. Relative density analyses were determined according to ISO 279 [35]. Refractive index analyses were carried out according to ISO 280 [36]. Measurements were made at 20 °C using a digital refractometer (A. Krüss Optronic GmbH. DR6000). The optical rotation value of lemon peel essential oils was determined according to ISO 592 [37] by a polarimeter device (Optical Activity Ltd. PolAAR 31).

2.5. Essential Oil Composition

The essential oil was analyzed by gas chromatography (Agilent 7890A)-mass spectrometry (Agilent 5975C)-flame ionization detector (GC-MS/FID device, Özek et al. [38]. Samples were diluted with hexane at a ratio of 1:50 to be analyzed. Essential oil component analysis of the samples was performed using a capillary column (HP Innowax Capillary; 60.0 m x 0.25 mm x 0.25 μm). GC-MS spectra were obtained using the following conditions: Carrier gas Helium; flow rate of 0.8 mL/min; and injection volume 1 μL with a split ratio of 50:1. The injector temperature was set at 250 °C. The column temperature program was set as 60 °C (10 minutes), 20 °C/minute from 60 °C to 250 °C, and 250 °C (10.5 minutes). In line with this temperature program, the total analysis time was 60 minutes. For the mass detector, scanning range (m/z) 35–500 atomic mass units and electron bombardment ionization 70 eV were used. WILEY and OIL ADAMS libraries were used to identify the components of the essential oil. Relative Retention Indices (RRI) of the compounds on the column were determined

relative to the retention times of a series of C8-C40 n-alkanes (Sigma, USA). Relative ratio amounts (%) of the determined components were calculated from FID chromatograms.

2.6. Statistical analyses

The research was carried out with three replications according to the randomized parcel trial design [39]. The analyses were carried out in two parallels, and the results were subjected to variance analysis (ANOVA) and the Duncan Multiple Comparison Test using the SAS package program. Results are given as mean ± standard error.

3. Results and discussion

The ANOVA and Duncan Multiple Comparison Test results for the fruit weights and peel ratios of the orange cultivars evaluated within the scope of the study are given in Table 2 and Table 3. While the cultivar had a statistically significant effect on the fruit weight and peel ratio of the oranges evaluated, the effects of harvest time and cultivar x harvest time remained insignificant. The cultivar with the highest fruit weight was Washington Navel, while the cultivar with the lowest was Moro. The fruit weights of the orange samples evaluated generally increased in parallel with the increase in harvest time. However, this difference remained statistically insignificant (Table 2).

Among the orange cultivars examined within the scope of the study, Moro had the highest peel ratio with 30.55%, and Valencia Late had the lowest peel ratio with 21.73%. Partial increases occurred in the fruit peel ratio with the advancement in harvest time, but these differences remained statistically insignificant (Table 2).

The volatile oil amounts of the sample peels of five orange cultivars evaluated in the project according to their harvest times and extraction applications are graphed in Figure 1. While the volatile oil rates of the orange samples showed significant differences according to the cultivars, the change remained insignificant according to the harvest time and cultivar x harvest time interaction (Table 2). Among the cultivars, the peel volatile oil amounts of Valencia Late (2.51%) and Navelina (2.55%) cultivars were higher than the others. The one with the lowest volatile oil rate among the cultivars was Moro (1.98%). When an evaluation was made according to the harvest times, the highest volatile oil amount was detected in Batem Fatihi, Navelina, Moro, and Valencia Late cultivars at the second harvest time, and in the Washington Navel cultivar at the first harvest time (Figure 1). When evaluated according to the methods of obtaining essential oil, the peel essential oil content of orange cultivars was higher with hydrodistillation (2.31%) than with cold press (0.48%). Geraci et al. [40] reported in their study on 12 orange cultivars that the amount of essential oil showed significant differences according to the cultivars as well as the extraction method. Ferrer et al. [41] reported that the amount of orange peel essential oil varies depending on the cultivar and the fruit maturity stage. In the study conducted by Bourgou et al. [42], it was determined that the amount of orange peel oil may vary according to the harvest period (a total of three periods). In the study, the lowest amount of essential oil was determined in the first harvest period, while the highest value was determined in the second harvest period. In the study conducted by Ferhat et al. [43] on 10 different citrus fruits, it was determined that the amount of essential oil obtained by hydrodistillation was considerably higher than the amount of oil obtained by cold pressing. In our study, it was revealed that there were some differences in this sense. When an evaluation was made on the amount of essential oil, it was seen that

it was appropriate to harvest all cultivars at the second harvest time except the Washington Navel cultivar (first harvest).

Table 2. Analysis of variance results for fruit weight, peel ratio, and essential oil content values of sweet oranges.

	Fruit weight		Peel ratio		Essential oil content	
	Statistic F	p-Value	Statistic F	p-Value	Statistic F	p-Value
Cultivar (C)	17.47	0.0001	4.74	0.0075	5.56	0.0014
Harvest time (HT)	1.18	0.3437	0.40	0.7568	2.07	0.0732
C×HT	0.53	0.8678	0.15	0.9991	1.08	0.4064
Coefficient of variation	15.3389		17.0473		15.7103	

Table 3. Duncan multiple comparision test results of fruit weight and peel ratio of the samples according to cultivar and harvesting time (mean ± standard error).

Cultivar	Harvest	Fruit weight (g/fruit)	Peel ratio (%)
Batem Fatihi	1	223.48 ± 15.390	22.79^{ab} ± 1.445
	2	250.06 ± 5.075	23.04^{ab} ± 3.635
	3	283.51 ± 20.305	22.95^{ab} ± 5.250
	4	296.37 ± 22.850	25.03^{ab} ± 3.810
	mean	263.36^{a} ± 12.62	23.45^{b} ± 1.473
Navelina	1	286.42 ± 35.735	25.15^{ab} ± 3.305
	2	295.33 ± 18.775	24.34^{ab} ± 4.940
	3	262.54 ± 83.700	25.92^{ab} ± 3.350
	4	301.91 ± 23.455	26.33^{ab} ± 2.510
	mean	286.55^{a} ± 18.97	25.43^{b} ± 1.404
W. Navel	1	307.45 ± 24.190	22.70^{ab} ± 1.465
	2	293.40 ± 22.060	23.90^{ab} ± 2.600
	3	298.56 ± 22.255	26.52^{ab} ± 3.135
	4	301.21 ± 21.960	26.72^{ab} ± 3.250
	mean	300.15^{a} ± 8.77	24.96^{b} ± 1.211
Valencia Late	1	167.56 ± 24.975	21.61^{b} ± 0.775
	2	199.44 ± 19.595	22.89^{ab} ± 1.640
	3	186.33 ± 1.040	22.04^{b} ± 2.155
	4	220.60 ± 7.775	20.38^{b} ± 0.070
	mean	193.48^{b} ± 9.57	21.73^{b} ± 0.633
Moro	1	168.90 ± 13.450	29.39^{a} ± 0.355
	2	155.64 ± 0.455	29.54^{a} ± 2.215
	3	202.42 ± 14.365	30.57^{a} ± 3.360
	4	184.47 ± 12.300	32.71^{a} ± 4.595
	mean	177.86^{b} ± 7.93	30.55^{a} ± 1.260

Different letters in the same column indicate significant differences between the means for cultivars at the $p < 0.05$ level.

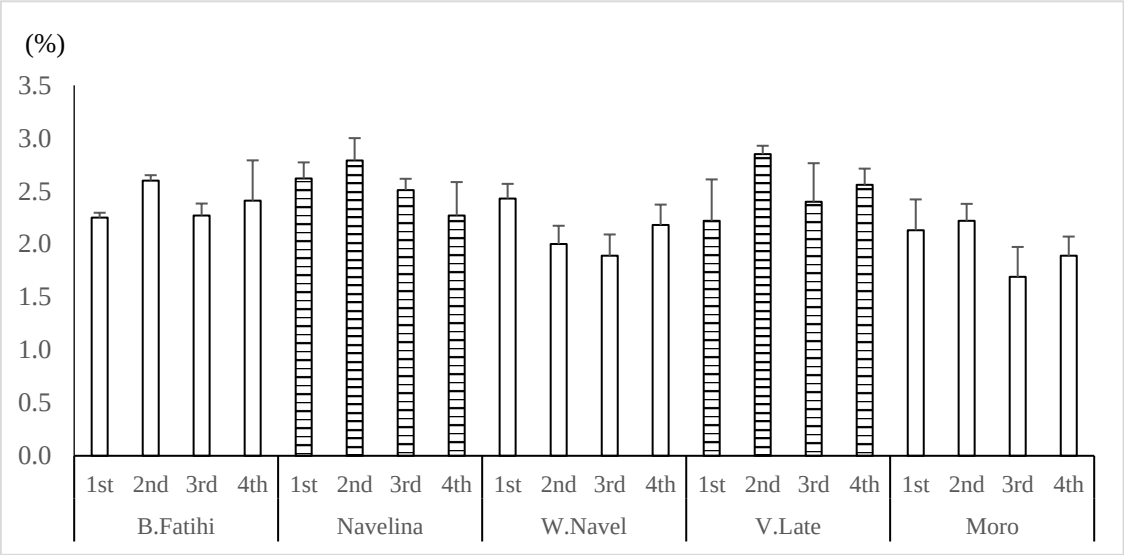

Figure 1. Essential oil contents of sweet orange cultivars according to harvest times (mean ± SE).

It was observed that there were some differences in the relative density, refractive index, and optical activity values of the orange peel oils evaluated within the scope of the research, especially according to the extraction method, harvest times, and cultivars. The variance analysis results of the relative density, refractive index, and optical activity values of the orange peel essential oils, according to the cultivar, harvest time, and extraction methods are given in Table 4, and the Duncan Multiple Comparison Test results are given in Table 5. The statistical evaluation data showed that the extraction method had a significant effect on the relative density, refractive index, and optical activity values of the orange peel essential oil, while the cultivar and harvest time factors were insignificant (Table 4). The average relative density value of the orange peel essential oils obtained by hydrodistillation was 0.8402, while the value obtained by cold pressing was 0.8444. The relative density value range for orange oil obtained by cold pressing in the European Pharmacopoeia is 0.842–0.850 [44], and it is reported as 0.842–0.850 in ISO standards [45]. In the study conducted by Xu et al. [46], the specific gravity value of Hamlin and Valencia peel essential oils obtained by cold pressing was reported as 0.843-0.848. In the study conducted by Li et al. [47], this value was reported as 0.8428 for orange peel essential oil. In our study, while the samples obtained by cold pressing were compatible with the limit values, the relative density values of the oils obtained by hydrodistillation remained below these values. It is thought that this situation may be due to the fact that the oils obtained by hydrodistillation consist only of volatile components.

When an evaluation was made between the extraction methods, it was determined that the refractive index values of the oils obtained by cold pressing were lower than the refractive index values of the samples obtained by the hydrodistillation method (Table 5). The refractive index value for the orange peel essential oil obtained by cold pressing was stated as 1.470–1.476 in the European Pharmacopoeia [44] and as 1.470–1.476 in ISO standards [45]. In the study conducted by the reserchers in [46] on Hamlin and Valenci cultivar orange peel oils, it was determined that the refractive index values of the samples obtained by the cold pressing method varied between 1.4718–1.4727. The researchers stated that values such as refractive index, specific gravity and optical activity can be used in the determination of adulteration in essential oils. In the study conducted by Javed et al. [48], the refractive index value of the orange peel essential oil

obtained by the hydrodistillation method was determined as 1.471. Within the scope of our study, it was observed that the refractive index values of the peel oils obtained by two different methods from five orange cultivars were compatible with the ISO [45] and European Pharmacopoeia [44] limit values and the literature values [46]. Augustyn et al. [49] determined the refractive index value of steam distilled Kisar sweet orange peel oil as 1.4651. Similar to our study, the higher density of the essential oil in cold pressing is associated with the higher refractive index [49].

Table 4. Analysis of variance results for relative density, refractive index, and optical activity values.

	Relative density		Refractive index		Optical activity	
	Statistic F	p-Value	Statistic F	p-Value	Statistic F	p-Value
Cultivar (C)	0.33	0.8577	0.33	0.8568	0.22	0.9269
Harvest time (HT)	0.73	0.5424	0.76	0.5250	1.76	0.1705
Isolation method (IM)	5.83	0.0204	148.86	0.0001	42.88	0.0001
C × HT	0.69	0.7510	0.48	0.9175	0.45	0.9332
C × IM	0.41	0.7980	0.36	0.8378	0.36	0.8328
HT × IM	0.75	0.5305	0.71	0.5523	0.05	0.9865
C × HT × IM	0.58	0.8459	0.12	0.9998	0.09	1.000
Coefficient of variation	0.8298		0.0398		1.9870	

Table 5. Relative density, refractive index, and optical activity values of orange peel essential oils according to cultivar, harvest time, and isolation method (mean ± standard error).

	Batem Fatihi	Navelina	W.Navel	V.Late	Moro
Relative density	0.8404 ± 0.0012	0.8428 ± 0.0023	0.8427 ± 0.0011	0.8419 ± 0.0013	0.8426 ± 0.0020
Refractive index	1.4731 ± 0.0003	1.4732 ± 0.0002	1.4730 ± 0.0003	1.4731 ± 0.0002	1.4731 ± 0.0003
Optical activity (°)	96.57 ± 0.595	96.96 ± 0.593	96.54 ± 0.593	97.02 ± 0.543	96.88 ± 0.413
	1.Harvest	2.Harvest	3.Harvest	4.Harvest	
Relative density	0.8426 ± 0.0015	0.8419 ± 0.0010	0.8404 ± 0.0013	0.8435 ± 0.0019	
Refractive index	1.4731 ± 0.0002	1.4730 ± 0.0002	1.4731 ± 0.0002	1.4733 ± 0.0002	
Optical activity (°)	96.95 ± 0.421	97.26 ± 0.352	97.01 ± 0.409	95.97 ± 0.652	
	Hydrodistillation		Cold-pressed		
Relative density	0.8402^{b} ± 0.0012		0.8440^{a} ± 0.0007		
Refractive index	1.4723^{b} ± 0.0001		1.4739^{a} ± 0.0001		
Optical activity (°)	98.20^{a} ± 0.234		95.39^{b} ± 0.272		

Different letters on the same line indicate a significant difference between the means at the $p < 0.05$ level.

The optical activity values of the samples varied between 93.93° and 99.63°. The optical activity values of orange peel oils varied within a narrow range according to the cultivars (Table 5). It is thought that this may be due to the similarity of the essential oil compositions of the orange cultivars. When an evaluation is made according to the harvest times, it can be said that there are generally small changes. According to the extraction methods, it was observed that there were significant differences in optical activity values as well as in the relative density and refractive index. It was determined that

the optical activity values of the essential oils obtained by the hydrodistillation method for orange cultivars were higher than the optical activity values of the samples obtained by cold pressing. It is thought that this result is due to the difference in the physico-chemical properties of the oils. The optical activity value of orange peel oil is limited between +94° and +99° in the European Pharmacopoeia [44]. The limit values of orange essential oil obtained by the cold pressing method are also reported in the same values in ISO standards [45]. In the study conducted by Javed et al. [48], the optical activity value of orange peel oil was determined as 89–91°. The findings determined within the scope of our research are in accordance with the limit values. However, it is higher than the values determined by Javed et al. [48]. In fact, the limonene value of the cultivars studied in the literature remained below 90%. It is thought that the detected difference is due to this composition difference.

Table 6. Essential oil composition (%) of the Batem Fatihi cultivar according to harvest time and the isolation method.

Compound	RRI	1. Harvest		2. Harvest		3. Harvest		4. Harvest	
		HD	CP	HD	CP	HD	CP	HD	CP
α-Pinene	1030	0.5	0.5	0.5	0.5	0.6	0.5	0.6	0.4
Sabinene	1132	0.3	0.3	0.3	0.3	0.3	0.3	0.4	0.5
δ-3-Carene	1156	0.2	0.2	0.2	0.2	0.3	0.2	0.2	0.2
β-Myrcene	1170	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
Limonene	1214	96.1	96.1	96.1	96.1	96.0	96.0	95.9	96.0
β-Phellandrene	1223	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3
Octanal	1302	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Decanal	1503	0.1	0.2	0.1	0.1	0.1	0.1	0.2	0.1
Linalool	1549	0.2	0.2	0.3	0.2	0.2	0.2	0.2	0.1
Geranial	1748	0.1	0.1	0.1	0.1	0.1	0.2	0.1	0.1

HD: Hydrodistillation, CP: Cold-pressed.

Table 7. Essential oil composition (%) of Navelina cultivar according to harvest time and the isolation method.

Compound	RRI	1. Harvest		2. Harvest		3. Harvest		4. Harvest	
		HD	CP	HD	CP	HD	CP	HD	CP
α-Pinene	1030	0.5	0.5	0.5	0.5	0.5	0.5	0.6	0.5
Sabinene	1132	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
δ-3-Carene	1156	0.2	0.2	0.3	0.3	0.3	0.3	0.2	0.3
β-Myrcene	1170	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
Limonene	1214	96.3	96.4	96.3	96.1	96.3	96.3	96.2	96.1
β-Phellandrene	1223	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3
Terpinolen	1298	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Octanal	1302	0.1	0.1	0.1	0.1	0.1	0.1	0.2	0.1
Decanal	1503	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Linalool	1549	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Undefined		0.1	0.0	0.1	0.3	0.0	0.1	0.2	0.2

HD: Hydrodistillation, CP: Cold-pressed.

Table 8. Essential oil composition (%) of Washington Navel cultivar according to harvest time and the isolation method.

Compound	RRI	1. Harvest		2. Harvest		3. Harvest		4. Harvest	
		HD	CP	HD	CP	HD	CP	HD	CP
α-Pinene	1030	0.6	0.5	0.5	0.5	0.6	0.5	0.6	0.5
Sabinene	1132	0.3	0.4	0.5	0.5	0.5	0.4	0.5	0.6
δ-3-Carene	1156	0.3	0.3	0.2	0.2	0.2	0.2	0.2	0.2
β-Myrcene	1170	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
Limonene	1214	95.7	95.8	95.5	95.4	95.8	95.9	95.3	95.4
β-Phellandrene	1223	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3
Octanal	1302	0.2	0.2	0.3	0.3	0.1	0.2	0.2	0.1
Decanal	1503	0.1	0.1	0.2	0.2	0.1	0.1	0.2	0.2
Linalool	1549	0.3	0.3	0.4	0.3	0.3	0.2	0.3	0.3
Geranial	1748	0.1	0.1	0.10	0.2	0.2	0.2	0.2	0.2
Undefined		0.1	0.0	0.13	0.3	0.1	0.0	0.3	0.3

HD: Hydrodistillation, CP: Cold-pressed.

Table 9. Essential oil composition (%) of Valencia Late cultivar according to harvest time and the isolation method.

Compound	RRI	1. Harvest		2. Harvest		3. Harvest		4. Harvest	
		HD	CP	HD	CP	HD	CP	HD	CP
α-Pinene	1030	0.6	0.5	0.6	0.5	0.6	0.5	0.5	0.5
Sabinene	1132	0.4	0.3	0.3	0.3	0.3	0.2	0.3	0.3
β-Myrcene	1170	2.0	2.0	2.0	2.0	2.0	2.0	2.0	1.9
Limonene	1214	96.2	96.2	95.8	95.9	95.9	96.3	96.0	96.2
β-Phellandrene	1223	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3
Octanal	1302	0.1	0.2	0.2	0.2	0.2	0.2	0.3	0.2
Decanal	1503	0.1	0.2	0.1	0.2	0.2	0.2	0.2	0.2
Linalool	1549	0.2	0.2	0.4	0.3	0.3	0.3	0.4	0.3
Valencene	1745	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Undefined		0.1	0.1	0.4	0.3	0.1	0.1	0.1	0.0

HD: Hydrodistillation, CP: Cold-pressed.

The average values of the compositions of the essential oils obtained from the peels of the orange cultivars evaluated within the scope of the project are given in Table 6, Table 7, Table 8, Table 9, and Table 10 for the cultivars Batem Fatihi, Navelina, Washington Navel, Valencia Late, and Moro, respectively. A total of 10 different components were detected in the essential oil of Batem Fatihi peel. The ratio of these components showed some numerical differences according to the harvest time and extraction method. The main component in Batem Fatihi peel essential oil was limonene. The ratio of this component was distributed in a narrow range of 95.9–96.1% in this cultivar. These data show that there was no significant difference in the ratio of this component according to the harvest time and extraction method for the Batem Fatihi cultivar. The second highest component in this cultivar was β-myrcene, which has a monoterpene structure like limonene. The amount of this component did not

show any significant difference according to the harvest time and extraction method. The ratio of other components detected in this orange cultivar remained below 1% (Table 6).

Table 10. Essential oil composition (%) of Moro cultivar according to harvest time and the isolation method.

Compound	RRI	1. Harvest		2. Harvest		3. Harvest		4. Harvest	
		HD	CP	HD	CP	HD	CP	HD	CP
α-Pinene	1030	0.6	0.5	0.6	0.6	0.6	0.6	0.6	0.5
Sabinene	1132	0.7	0.7	0.6	0.6	0.7	0.6	0.7	0.6
δ-3-Carene	1156	0.1	0.2	0.2	0.3	0.1	0.2	0.1	0.1
β-Myrcene	1170	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
Limonene	1214	94.9	94.9	95.2	95.2	95.0	95.2	95.1	95.2
β-Phellandrene	1223	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3
Octanal	1302	0.3	0.2	0.1	0.1	0.1	0.1	0.2	0.1
Decanal	1503	0.2	0.2	0.1	0.1	0.1	0.1	0.2	0.2
Linalool	1549	0.6	0.5	0.4	0.3	0.5	0.4	0.4	0.4
Geranial	1748	0.2	0.2	0.3	0.3	0.3	0.3	0.3	0.4
Undefined		0.2	0.3	0.2	0.4	0.3	0.2	0.1	0.2

HD: Hydrodistillation, CP: Cold-pressed.

Similar essential oil components were detected in the second orange cultivar evaluated within the scope of the research, the Naveline cultivar, and a significant portion of the essential oil components of this cultivar consists of limonene, a monoterpene structure with various functional properties. The ratio of this component varied between 96.1–96.4% in this cultivar. The second component in this cultivar was β-myrcene, and the ratio of this component was 2.0% for all samples. It was seen that the ratio of other components was similar to other orange cultivars. Unlike the Batem Fatihi cultivar, terpinolene component was detected instead of geranial in this cultivar. However, the ratio of this component remained at a very low level (Table 7). Although some numerical differences were detected in the essential oil compositions of Washington Navel, Valencia Late, and Moro cultivars, these were at very low levels in proportion. Limonene is again the main component in these cultivars and it was distributed between 95.3–95.9% in Washington Navel, 95.8–96.3% in Valencia Late, and 94.9–95.2% in Moro cultivar. As in the other two orange cultivars, it was determined that the second most important component in Washington Navel, Valencia Late, and Moro cultivars was β-myrcene. In these cultivars, the ratio of this component was similar to Batem Fatihi and Navelina.

The two major components of orange samples, limonene and β-myrcene, were statistically evaluated according to cultivar, harvest time, and extraction method (Table 11, Table 12). β-Myrcene contents of the samples showed significant changes according to cultivar ($p < 0.01$) and harvest time ($p < 0.05$), but statistically insignificant changes according to extraction method and interactions (Table 11). The cultivar with the highest β-myrcene content among the cultivars was Navelina, while the lowest was Valencia Late. It was determined that the β-myrcene rate decreased with the advancement of harvest time. However, the differences were numerically quite low (Table 12). Limonene contents in orange peel essential oils also showed statistically significant changes among cultivars ($p < 0.01$), but insignificant changes according to harvest time, extraction method, and interactions. Among the cultivars, Navelina had the highest limonene content, followed by Valencia Late, Batem Fatihi,

Washington Navel, and Moro cultivars, respectively. However, these differences occurred within a very narrow range (Table 12).

In the study conducted by Ferrer et al. [41] on Cara Cara Navel and Madam Vinous cultivars from the fruit formation stage to the later stages of maturity, it was determined that there were significant changes in the peel essential oil composition until the maturity stage was reached. It was determined that limonene, the main component of the peel essential oil, varied between 25–95% throughout all stages. However, no significant change was detected in the essential oil composition of the samples between the cultivars. In the study conducted by Li et al. [47], the ratio of limonene, determined as the main component, in the peel oil was reported as 78.30%. In the study conducted by Lin et al. [50] on the Newhall cultivar, it was determined that the limonene and β-myrcene contents of the peel essential oil obtained by hydrodistillation were 88.25% and 1.90%, respectively. Viuda-Martos et al. [51] similarly determined the major components limonene and β-myrcene as 94.9% and 1.16%, respectively. Taktak et al. [52] investigated the effects of different extraction methods on the yield and composition of essential oils from the peel of some citrus species, including orange. In the study, some differences were detected in the composition of the peel essential oil according to the extraction methods, and it was determined that limonene in the composition of the orange peel essential oil obtained by hydrodistillation was 86.7%. Brahmi et al. [53] also investigated the effects of hydrodistillation and microwave-assisted hydrodistillation methods on the composition of the essential oil of Valencia orange. As a result of the study, it was determined that the extraction method was effective on the essential oil composition as well as the yield of the essential oil. While the limonene ratio in the sample obtained by hydrodistillation was 92.74%, it was determined as 89.86% in the microwave-assisted hydrodistillation method. In a study conducted by Wolfenbüttel et al. [54], it was determined that limonene, the main component in peel oils obtained by hydrodistillation and cold pressing from oranges obtained from different regions in Brazil, varied between 67.7–78.3%. Researchers also reported that this component in orange peel essential oil varied between 78.5–97% in studies conducted in different countries. In another evaluation conducted on long-term data, limonene and β-myrcene ranges for orange peel essential oil were reported as 85.16–96.80% and 0.93–2.05%, respectively [55]. In the study conducted by Ferreira et al. [56], it was determined that limonene, the main component of the peel oil obtained by hydrodistillation and cold pressing method, was in the range of 96.1-96.6% and β-myrcene was 1.9%. Limit values are given for α-pinene, β-pinene, sabinene, β-myrcene, limonene, octanal, decanal, linalool, neral, valensene, and geranial in the European Pharmacopoeia for orange peel oils. Reference values for limonene and β-myrcene, which are the major components, are stated as 92–97%, 1.7–2.5%, respectively [44]. Limit values for limonene and β-myrcene are also reported as 93–96% and 1.5–3.5%, respectively, in ISO standards [45]. It was observed that the five cultivars evaluated within the scope of our study were within these limit values.

Table 11. Analysis of variance results for β-myrcene and limonene contents.

	β-Myrcene		Limonene	
	Statistic F	p-Value	Statistic F	p-Value
Cultivar (C)	3.46	0.0162	20.65	0.0001
Harvest time (HT)	3.00	0.0417	0.40	0.7565
Isolation method (IM)	0.33	0.5669	0.63	0.4310
C × HT	0.57	0.8532	0.47	0.9223
C × IM	0.13	0.9726	0.15	0.9605
HT × IM	0.33	0.8013	0.14	0.9334
C × HT × IM	0.68	0.7595	0.07	1.000
Coefficient of variation	1.9341		0.4254	

Table 12. Duncan multiple comparision test results of β-myrcene and limonene contents (%) of orange peel essential oil according to cultivar, harvest time, and isolation method (mean ± standard error).

	B.Fatihi	Navelina	W.Navel	V.Late	Moro
β-Myrcene	$2.01^{a} \pm 0.006$	$2.03^{a} \pm 0.011$	$2.01^{a} \pm 0.006$	$1.98^{b} \pm 0.011$	$2.0^{ab} \pm 0.009$
Limonene	$96.03^{a} \pm 0.051$	$96.24^{a} \pm 0.032$	$95.59^{b} \pm 0.092$	$96.06^{a} \pm 0.063$	$95.09^{c} \pm 0.135$
	1.Harvest	2.Harvest	3.Harvest	4.Harvest	
β-Myrcene	$2.02^{a} \pm 0.009$	$2.01^{a} \pm 0.007$	$1.99^{b} \pm 0.007$	$1.99^{b} \pm 0.010$	
Limonene	95.84 ± 0.121	95.76 ± 0.110	95.87 ± 0.127	95.75 ± 0.120	
	Hydrodistillation		Cold-pressed		
β-Myrcene	2.01 ± 0.006		2.00 ± 0.006		
Limonene	95.77 ± 0.085		95.84 ± 0.082		

Different letters on the same line indicate a significant difference between the means at the $p < 0.05$ level.

4. Conclusions

As a result of the research; when the analysis findings obtained were evaluated, it was determined that the orange peel essential oil ratio showed significant differences according to the cultivar and harvest time. It was observed that the relative density, refractive index, and optical activity properties showed significant differences especially according to the essential oil extraction method. It was determined that the essential oil composition could show partial differences according to the cultivar, harvest time, and extraction method. However, these differences generally remained insignificant at a statistical level. Limonene and β-myrcene were the major components in all the evaluated samples. According to the findings obtained as a result of the study, it was shown that all of these cultivars can be used in the production of orange peel oils. The cultivars within the scope of our study attracted attention with their generally high limonene content according to international standards (European Pharmacopoeia and ISO standards). Cold press application is a widely preferred method for orange peel essential oils on the industrial scale. On the other hand, an evaluation was made on the basis of two isolation methods, especially in terms of limonene, which is the functional component of citrus oils, it was determined that the production of orange peel oils by the hydrodistillation method did not cause any negative effects in terms of limonene content. These data show that the essential oils

obtained by hydrodistillation can be used in many areas, especially in the food industry and cosmetics industry.

Use of AI tools declaration

The authors declare they have not used Artificial Intelligence [AI] tools in the creation of this article.

Acknowledgments

This study was prepared using the findings of the project no. TAGEM/TBAD/B/21/A7/P6/2370, supported by the General Directorate of Agricultural Research and Policies [TAGEM], Republic of Türkiye Ministry of Agriculture and Forestry.

Conflict of interest

The authors declare no conflict of interest.

Authors contributions

Plant authority, E.T.; conceptualization, M.G.; methodology, M.G.; software, M.G. and A.M.G.; validation, B.B., M.G. and A.M.G.; formal analysis, B.B. and M.G.; investigation, M.G. and O.Ç., D.Y.T., H.T.; resources, B.B. and M.G.; data curation, B.B., M.G., O.Ç., D.Y.T., H.T., E.T. and A.M.G.; writing – original draft preparation, M.G., B.B. O.Ç., D.Y.T., H.T., E.T.; writing – review and editing, B.B., M.G. and A.M.G.; visualization, B.B., M.G. and A.M.G.; supervision, B.B., M.G. and A.M.G.; project administration, B.B., M.G. and A.M.G.; funding acquisition, M.G. All authors have read and agreed to the published version of the manuscript

References

1. Raveau R, Fontaine J, Sahraoui AL (2020) Essential oils as potential alternative biocontrol products against plant pathogens and weeds: A review. *Foods*. 9: 365. https://doi.org/10.3390/foods9030365
2. Sharma A, Gumber K, Gohain A, et al. (2023) Importance of essential oils and current trends in use of essential oils [aroma therapy, agrofood, and medicinal usage]. In: Nayik GA, Ansari MJ (Eds.), *Essential oils extraction, characterization, and applications,* London, UK: Academic Pess, 53–84. https://doi.org/10.1016/B978-0-323-91740-7.00002-5
3. Devi MP, Chakrabarty S, Ghosh SK, et al. (2015) Essential oil: Its economic aspect, extraction, importance, uses, hazards and quality. In: Sharangi AB, Datta S (Eds.), *Value addition of horticultural crops: Recent trends and future directions,* Londo, UK: Springer, 269–278. https://doi.org/10.1007/978-81-322-2262-0_15

4. Khan S, Sahar A, Tariq T, et al. (2023) Essential oils: Plants physiology, the chemical composition of the oil, and the natural variation of the oils [chemotaxonomy and environmental effects, etc.]. In: Nayik GA, Ansari MJ (Eds.), *Essential oils extraction, characterization, and applications,* London, UK: Academic Pess, 1–37. https://doi.org/10.1016/B978-0-323-91740-7.00016-5
5. Gavahian M, Yan-Hwa Chu YH, Amin Mousavi Khaneghah AM (2019) Recent advances in orange oil extraction: an opportunity for the valorisation of orange peel waste a review. *Int J Food Sci Technol* 54: 925–932. https://doi.org/10.1111/ijfs.13987
6. Yaqoob M, Aggarwal P, Aslam R, et al. (2020) Extraction of bioactives from citrus. In: Boddula IR, Asiri AM (Eds.), *Green sustainable process for chemical and environmental engineering and science,* Amsterdam, Netherlands: Elsevier, 357–377. https://doi.org/10.1016/B978-0-12-817388-6.00015-5
7. Manakas P, Balafoutis AT, Kottaridi C, et al. (2024) Sustainability assessment of orange peel waste valorization pathways from juice industries. *Biomass Convers Biorefin* 15: 6525–6544. https://doi.org/10.1007/s13399-024-05626-x
8. Kant R, Kumar A (2022) Reviews on essential oil extraction from aromatic and medicinal plants: Techniques, performance and economic analysis. *Sustainable Chem Pharm* 30: 100829. https://doi.org/10.1016/j.scp.2022.100829
9. Turkish Statistical Institute [TÜİK] (2024a) Bitkisel Üretim İstatistikleri. Available from: http://tuikapp.tuik.gov.tr/bitkiselapp/bitkisel.zul.
10. Sawamura M (2011) *Citrus essential oils flavor and fragrance*. Singapore: John Wiley & Sons, 398p. https://doi.org/10.1002/9780470613160
11. Roussos PA (2016) Orange [*Citrus sinensis* [L.] Osbeck]. In: Simmonds MSJ, Preedy VR (Eds.), *Nutritional compoition of fruit cultivars,* London, UK: Academic Press, 469–496. https://doi.org/10.1016/B978-0-12-408117-8.00020-9
12. Chemat F (2010) Techniques for oil extraction. In: Dugo G, Mondello L (Eds.), *Citrus oils composition, advanced analytical techniques, contaminants, and biological activity,* London, UK: CRC Press, Taylor & Francis Group, 9–36. https://doi.org/10.1002/9780470613160.ch2
13. Gölükcü M, Bozova B, Tokgöz H, et al. (2024) Effects of cultivar, harvesting time and isolation techniques on the essential oil compositions of some lemon cultivars. *AIMS Agric Food* 9: 904–920. https://doi.org/10.3934/agrfood.2024049
14. Bozova B, Gölükcü M, Giuffrè AM (2024) The effect of different hydrodistillation times on the composition and yield of bergamot (*Citrus bergamia* Risso) peel essential oil and a comparison of the cold-pressing method. *Flavour Fragrrance J* 39: 263–270. https://doi.org/10.1002/ffj.3789
15. Gioffrè G, Ursino D, Labate MLC, et al. (2020) The peel essential oil composition of bergamot fruit (*Citrus bergamia*, Risso) of Reggio Calabria (Italy): A review. *Emirates J Food Agric* 32: 835–845. https://doi.org/10.9755/ejfa.2020.v32.i11.2197
16. Giuffré AM, Nobile R (2020) *Citrus bergamia,* Risso: The peel, the juice and the seed oil of the bergamot fruit of Reggio Calabria (South Italy). *Emirates J Food Agric* 32: 522–532. https://doi.org/10.9755/ejfa.2020.v32.i7.2128
17. Gonzalez-Mas MC, Rambla JL, López-Gresa MP, et al. (2019) Volatile compounds in citrus essential oils: A comprehensive review. *Front Plant Sci* 10: 1–18. https://doi.org/10.3389/fpls.2019.00012
18. Singh B, Singh JP, Kaur A, et al. (2021) Insight into the chemical composition and bioactives of citrus peel essential oils. *Food Res Int* 143: 1–19. https://doi.org/10.1016/j.foodres.2021.110231

19. Bora H, Kamle M, Mahato DK, et al. (2020) Citrus essential oils [CEOs] and their applications in food: An overview. *Plants* 9: 1–25. https://doi.org/10.3390/plants9030357
20. Palazzolo E, Laudicina VA, Germana MA (2013) Current and potential use of citrus essential oils. *Curr Org Chem* 17: 3042–3049. https://doi.org/10.2174/13852728113179990122
21. Agarwal P, Sebghatollahi Z, Kamal M, et al. (2022) Citrus essential oils in aromatherapy: Therapeutic effects and mechanisms. *Antioxidants* 11: 2374. https://doi.org/10.3390/antiox11122374
22. Türkmenoğlu A, Özmen D (2023) Deterpenation techniques of citrus essential oils and the role of oxyterpenes in food industry. *Flavour Fragrance J* 38: 407–415. https://doi.org/10.1002/ffj.3758
23. Bousbia N, Vian MA, Ferhat MA, et al. (2009) A new process for extraction of essential oil from Citrus peels: Microwave hydrodiffusion and gravity. *J Food Eng* 90: 409–413. https://doi.org/10.1016/j.jfoodeng.2008.06.034
24. FAOSTAT (2024) FAO Statistical Database. Available from: http://faostat3.fao.org/home/E.
25. Turkish Statistical Institute [TÜİK] (2024) Dış Ticaret İstatistikleri. Available from: https://biruni.tuik.gov.tr/medas.
26. Fisher K, Phillips C (2008). Potential antimicrobial uses of essential oils in food: is citrus the answer? *Trends Food Sci Technol* 19: 156–164. https://doi.org/10.1016/j.tifs.2007.11.006
27. Hosni K, Zahed N, Chrif R, et al. (2010) Composition of peel essential oils from four selected Tunisian citrus species: Evidence for the genotypic influence. *Food Chem* 123: 1098–1104. https://doi.org/10.1016/j.foodchem.2010.05.068
28. Shahidi F, Zhong Y (2005) Citrus oils and essences. In: Shahidi F (Ed.), *Bailey's Industrial Oil and Fat Products,* Six Edition, USA: John Wiley & Sons, Inc., 49–66. https://doi.org/10.1002/047167849X.bio051
29. Chiralt A, Martinez-Monzo J, Chafer T, et al. (2002) Limonene from citrus. In: Shi J, Mazza G, Le Maguer M (Eds.), *Functional Foods: Biochemical and Processing Aspects,* Boca Raton, FL: CRC Press, 163–180.
30. Bacanlı M, Başaran AA, Başaran N (2018) Effects and Usage of a Citrus Compound, Limonene [Chapter 32]. In: Watson RR, Preedy VR, Zibadi S (Eds.), *Polyphenols: Prevention and Treatment of Human Disease,* London, UK: Academic Press, Elsevier, 419–424. https://doi.org/10.1016/B978-0-12-813008-7.00032-1
31. Mahato N, Sharma K, Koteswararao R, et al. (2019) Citrus essential oils: Extraction, authentication and application in food preservation. *Crit Rev Food Sci Nutr* 59: 611–625. https://doi.org/10.1080/10408398.2017.1384716
32. Park MK, Cha JY, Kang MC, et al. (2024) The effects of different extraction methods on essential oils from orange and tangor: From the peel to the essential oil. *Food Sci Nutr* 12: 804–814. https://doi.org/10.1002/fsn3.3785
33. International Organisation for Standardization [ISO] (2008) ISO 6571-Spices, condiments and herbs-Determination of volatile oil content (hydrodistillation method).
34. Kirbaslar FG, Kirbaslar SI, Dramur U (2001) The compositions of Turkish bergamot oils produced by cold-pressing and steam distillation. *J Essent Oil Res* 13: 411–415. https://doi.org/10.1080/10412905.2001.9699710
35. International Organisation for Standardization (1998) ISO 279-Essential oils-Determination of relative density at 20 °C-Reference method.

36. International Organisation for Standardization (1998) ISO 280-Essential oils-Determination of refractive index.
37. International Organisation for Standardization (1998) ISO 592-Essential oils-Determination of optical rotation.
38. Özek G, Demirci F, Özek T, et al. (2010) Gas chromatographic–mass spectrometric analysis of volatiles obtained by four different techniques from *Salvia rosifolia* Sm., and evaluation for biological activity. *J Chromatogr A* 1217: 741–748. https://doi.org/10.1016/j.chroma.2009.11.086
39. Düzgüneş O, Kesici T, Kavuncu O, et al. (1987) Araştırma ve Deneme Metotları. *Ankara Üniversitesi, Ziraat Fakültesi Yayınları*. No: 1021, Ankara, Türkiye, 229 p.
40. Geraci A, Di Stefano V, Di Martino E, et al. (2017) Essential oil components of orange peels and antimicrobial activity. *Nat Prod Res* 31: 653–659. https://doi.org/10.1080/14786419.2016.1219860
41. Ferrer V, Paymal N, Quinton C, et al. (2022) Investigations of the chemical composition and aromatic properties of peel essential oils throughout the complete phase of fruit development for two cultivars of sweet orange [*Citrus sinensis* [L.] Osb.]. *Plants* 11: 2747. https://doi.org/10.3390/plants11202747
42. Bourgou S, Rahali FZ, Ourghemmi I, et al. (2012) Changes of peel essential oil composition of four Tunisian citrus during fruit maturation. *Sci World J* 2012: 1–10. https://doi.org/10.1100/2012/528593
43. Ferhat MA, Boukhatem MN, Hazzit M, et al. (2016) Cold Pressing, hydrodistillation and microwave dry distillation of citrus essential oil from Algeria: A comparative study. *Electron J Biol* S1: 30–41.
44. Anonymous, (2008) Sweet orange aurantii dulcis aetheroleum. *European Pharmacopeia*. 3009–3010.
45. Anonymous (2019) Essential oil of sweet orange expressed [*Citrus sinensis* [L.]]. International Standards, ISO 3140.
46. Xu BM, Sims CA, Etxeberria E, et al. (2017) Physicochemical and sensory properties of cold pressed oils from Florida Hamlin and Valencia Oranges affected by Huanglongbing. *J Food Sci* 82: 2158–2166. https://doi.org/10.1111/1750-3841.13814
47. Li Y, Liu S, Zhao C, et al. (2022) The chemical composition and antibacterial and antioxidant activities of five Citrus essential oils. *Molecules* 27: 44. https://doi.org/10.3390/molecules27207044
48. Javed S, Javaid A, Nawaz S, et al. (2014) Phytochemistry, GC-MS analysis, antioxidant and antimicrobial potential of essential oil from five citrus species. *J Agric Sci* 6: 201–208. https://doi.org/10.5539/jas.v6n3p201
49. Augustyn GH, Matjora A, Lawalata VN, et al. (2021) Isolation and identification of essential oil made from Kisar sweet orange peels. *Trop Small Island Agric Manag* 1: 1–11. https://doi.org/10.30598/tsiam.2021.1.1.1
50. Lin X, Cao S, Sun J,et al. (2021) The chemical composition, and antibacterial and antioxidant activities of four types of citrus essential oils. *Molecules* 26: 3412. https://doi.org/103390/molecules26113412
51. Viuda-Martos M, Ruiz-Navajas Y, Fernández-López J, et al. (2009) Chemical composition of mandarin (*C. reticulata* L.), grapefruit (*C. paradisi* L.), lemon (*C. limon* L.) and orange (*C. sinensis* L.) essential oils. *J Essent Oil Bear Plants* 12: 236–243. https://doi.org/10.1080/0972060X.2009.10643716

52. Taktak O, Youssef SB, Vian MA, et al. (2021) Physical and chemical influences on different extraction techniques for essential oil recovery from *Citrus sinensis* peels. *J Essent Oil Bear Plants* 24: 290–303. https://doi.org/10.1080/0972060X.2021.1925596
53. Brahmi F, Mokhtari O, Legssyer B, et al. (2021) Chemical and biological characterization of essential oils extracted from citrus fruits peels. *Mater Today: Proc* 45: 7794–7799. https://doi.org/10.1016/j.matpr.2021.03.587
54. Wolffenbüttel AN, Zamboni A, Dos Santos MK, et al. (2015) Chemical components of citrus essential oils from Brazil. *Nat Prod J* 5: 14–27. https://doi.org/10.2174/2210315505011504140953331
55. Dugo G, Controneo A, Bonaccorsi I, et al. (2011) Composition of the volatile fraction of citrus peel oils. In: Dugo G, Mondello L (Eds.), *Citrus oils composition, advanced analytical techniques, contaminants, and biological activity,* London, UK: CRC Press, Taylor & Francis Grou, 1–161. https://doi.org/10.1201/b10314
56. Ferreira G, Sobral L, Barreto DW, et al. (2020) Thermal and compositional analysis of orange essential oil obtained from citrus ındustry waste. *J Adv Therm Sci Res* 7: 48–55. https://doi.org/10.15377/2409-5826.2020.07.6